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JULY 1947

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BY

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I. C. 7412,
July 1947.

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QUANTITATIVE ESTIMATION OF POTASH AND SODA FELDSPARS IN PEGMATITE ROCK BY MEANS OF CHEMICAL COLORATION: A REVIEW OF SELECTED LITERATURE[1]

By Gerald A. Munson[2] and E. P. Barrett[3]

Rapid and accurate estimation of the proportions of mineral in rock mixtures is essential in mineral-dressing research and industrial operation.

Estimation of the proportions of potash and soda feldspars in ground pegmatite material by casual observation is almost impossible and may be tedious or even difficult by petrographic examination. Chemical analysis may not be an accurate measure, as pegmatite rock may contain the following minerals:

Mineral	Theoretical composition
Quartz	SiO_2
Potash feldspar	$KalSi_3O_8$
Plagioclase	$NaAlSi_3O_8 - CaAl_2Si_2O_8$.
Muscovite	$Kal_3Si_3O_{10}(OH)_2$
Biotite	$K(Mg,Fe)_3AlSi_3O_{10}(OH)_2$
Tourmaline	$(Na,Ca)(Fe,Li,Mg)_3B_3Al_3(AlSi_2O_9)_3(O,OH,F)_4$
Apatite	$Ca_5(F,Cl)(PO_4)_3$.

Metallurgists and petrographers have used selective staining of one or more minerals in rock mixtures to identify the various minerals and to afford a method by which the proportions may be estimated by a gain count under a microscope.

1/ The Bureau of Mines will welcome reprinting of this paper, provided the following acknowledgment is used: "Reprinted from Bureau of Mines Information Circular 7412."

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Johannsen and Merritt[4/] developed a method for recognizing minerals and determining their proportions in crushed rock.

About 0.5 gram of grains of uniform size (minus 80 plus 100 mesh) is placed in a lead dish containing 50 percent hydrofluoric acid and allowed to stand 7 minutes. Too long etching dissolves potash feldspar, whereas too short etching does not attack plagioclase. The acid produces a gelatinous film on the feldspars, whereas the quartz is virtually unaffected in the short time. The material is washed with a fine jet of water, after which a strong solution of gentian violet is added and allowed to stand for 10 minutes. The plagioclase shows an opaque stained mantle, the potash feldspar is corroded but transparent, and the quartz is unaffected.

The method may be made quantitative by counting the grains of the various minerals by means of a grating micrometer eyepiece on the microscope.

Grains of uniform size are necessary if the grain count is to have quantitative value.

About 80 magnification is suitable for viewing minus 80 plus 100 mesh particles.

Microcline and orthoclase are more readily decomposed than plagioclase. The time allowed for the acid to react is governed by the state of alteration. In some samples the weathering may have proceeded so far that the acid decomposes the potash feldspar before it attacks plagioclase sufficiently to be of practical value.

Stains other than gentian violet may be used, although this stain was used satisfactorily.

All of the grains treated with hydrofluoric acid and stained should be placed on the slide to avoid differential separation of particles of different physical properties. With medium magnification, about 80 grains in a field is convenient. There should be no crowding or overlapping of grains.

While studying the separation of quartz from feldspar by flotation, Engel[5/] developed a method by which the feldspars are distinctly stained, whereas the quartz is not. The proportions of stained and unstained particles in the products indicate the degree of separation achieved.

A few grains of a representative sample of the material are placed in a beaker and covered with warm 1:1 hydrochloric acid. The material is digested for 5 minutes, washed until free of acid, and dried on a sand bath. When dry, the material is covered with a 2-percent solution of safranine O (2 grams of the dye in 30 ml. of ethyl alcohol and 70 ml. of distilled water) and gently

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4/ Johannsen, Albert, and Merritt, C. A., The Recognition of Minerals and the Determination of Their Proportions in Crushed Rocks: Jour. of Geol., vol. 34, 1926, pp. 462-465.

5/ Engel, A. L., A Staining Method for the Differentiation Between Feldspar and Quartz: Bureau of Mines Rept. of Investigations 3370, 1938, pp. 69-70.

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warmed on a hot plate, being allowed to stand 15 minutes or more. The solution is decanted, and the material is thoroughly washed with distilled water. It is necessary to tumble the grains while washing to remove all traces of the free dye before drying on the sand bath.

To establish the consistency of this staining method, a portion of ore was crushed to minus 10 mesh and sized. Each size was treated, and the stained and unstained grains were separated under a binocular ore-dressing microscope. Engel states:

> It is not practical to separate the grains on material finer than 65 mesh. The relative proportion of feldspar and quartz in the plus 65 mesh sizes was about 60 percent feldspar, 40 percent quartz, and since the plus 65 mesh portions amounted to 84 percent of the original sample it is believed that size of particle causes no inconsistency in staining.
>
> This method is considered satisfactory.

Gabriel and Cox[6/] developed a method in which small samples of crushed material were subjected to etching in hydrofluoric acid fumes to decompose the surface of the mineral constituents. This treatment required 10 or 12 minutes at temperatures of 60° to 70° C. and left a faint white coating on the surface of the grains. The grains were then carefully immersed in a solution containing 15 ml. glacial acetic acid, 25 ml. water, 12.5 gm. $Co(NO_3.6\ H_2O$, and 20 gm. $NaNO_2$. The sample was decanted free of the reagent and washed with water by decantation. The dried grains were ready for microscopic examination.

After this treatment, the potash feldspars are intensely yellow, quartz is transparent, the plagioclases are white and opaque to transmitted light, and the micas remain white or black, depending upon variety.

If the biotite is decomposed by weathering, it yields a yellow precipitate, and the original black will show through the yellow and give the grain a decided green color. The micas, especially muscovite, are not usually decomposed by the treatment.

If desired, the identification of plagioclases can be further enhanced by the use of a dye such as eosin, which stains them but leaves the potash feldspars predominately yellow.

Russell[7/] simplified the procedure developed by Gabriel and Cox. He mounted a small sample of crushed grains on a slide glass covered with a film of Canada balsam or kollolith. The grains were covered with a few drops of hydrofluoric acid, which was allowed to remain for about 1 minute, and was then removed by washing with water. The acid produces a gelatinous film of

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6/ Gabriel, A., and Cox, E. P., A Staining Method for the Quantitative Determination of Certain Rock Minerals: American Mineralogist, vol. 14, 1929, pp. 290-292.

7/ Russell, R. Dana, Frequency Percentage Determinations of Detrital Quartz and Feldspar: Jour. Sedimentary Petrology, vol. 5, No. 3, December 1935, pp. 109-114.

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aluminum fluosilicate on the feldspars and other aluminous minerals but leaves the quartz clear. The acid must be washed off gently, otherwise the film on the feldspar grains will be removed. The washed grains were treated according to the procedure outlined by Gabriel and Cox.

It requires about 15 minutes to prepare a slide and 15 to 20 minutes to make the grain count.

Kruger8/ slightly modified the methods described by Gabriel and Cox and by Russell. He used dilute hydrofluoric acid better to control the etching of the mineral particles; otherwise, his method was the same.

COBALTINITRITE STAINING METHOD

The cobaltinitrite staining method has been used in the pegmatite research laboratory of the Bureau of Mines, Rapid City, S. Dak., on samples of crushed raw pegmatite and on products of mineral-dressing tests.

Products from flotation operations must be washed in ether or alcohol to remove oil film.

A small sample of closely sized material is placed in a dish resistant to hydrofluoric acid and covered with 1:1 hydrofluoric acid. After 1 to 5 minutes, the acid is removed by decantation. The free acid is gently washed from the grains, after which they are transferred to a 50-ml. beaker containing 0.1 to 0.2 gram sodium cobaltinitrite powder and 25 ml. distilled water. The solution is stirred, allowed to stand for about 5 minutes, and decanted. The grains are washed well by gently tumbling in water. After the water is removed by decantation, the grains are dried.

The potash feldspar is colored a bright canary yellow, the quartz is unaffected and transparent, and the plagioclases are white and opaque under reflected light. The micas, tourmaline, and spodumene appear to be unaffected and can be identified by their color or crystal habit. Accurate estimates of the proportions of each mineral can be made by particle counts under an ordinary microscope.

This method may be applied to unsized material to obtain approximate proportions of contained potash feldspar quickly. No true quantitative estimates can thus be obtained, but the treatment makes obvious the mineral ratio and may circumvent unnecessarily detailed study. Hand specimens likewise react to the treatment.

NOTES

The heat of reaction of the hydrofluoric acid upon the mineral particles increases the temperature of the solution and the rate of reaction. Care must be exercised so that the fine particles are not taken into solution or the quartz is not etched so as to confuse it with the plagioclases. Normally, 5

8/ Kruger, Frederick, C., A Sedimentary and Petrographic Study of Certain Glacial Drifts of Minnesota: Am. Jour. Science, 5th sec., vol. 34, No. 203.

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minutes is adequate, but less time or more dilute acid may be used on minus 100-mesh material.

Potassium cobaltinitrite is soluble and decomposes in hot water. The precipitation of this compound must be accomplished in cold solutions, preferably about 50° F. The coated particles must be washed carefully with cold water to prevent dislodgement of the precipitate. Decantation of the wash water should be as complete as possible before drying.

Stock solutions of sodium cobaltinitrite tend to deteriorate in a short time through decomposition of the compound. The use of the powder is convenient, and there is no decomposition if the container is kept stoppered.

The treatment with sodium cobaltinitrite involves a chain reaction, the ultimate result of which may be expressed in the following empirical equation:

$$3K^+ + CO(NO_2)_3\ 3NaNO_2 + H_2O \longrightarrow$$

$$K_3CO(NO_2)_6H_2O + 3Na^+$$

The solution becomes cloudy with potassium cobaltinitrite during this reaction, and the solution must reach saturation before complete staining is accomplished. If the treatment is prolonged, all the potassium may be dissolved from the mineral surface.

Larger specimens treated by the process display something of the physical structure of the rock, in that transparent ridges of quartz are evident. The ridges are separated by etched grooves filled with the yellow crystals of the precipitate or the white, spongy material characteristic of the treated plagioclase. Fractures in the potash feldspar filled with plagioclase or quartz are easily discernible, and frequently blobs of transparent quartz or the white opaque plagioclases are seen.

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